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Fresh and Frozen Poultry Product Regulation

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Report Highlights:

The following is an UNOFFICIAL translation of the People's Republic of China Fresh and Frozen Product Regulation (GB 1689-2002) and should be used as a guide only. Exporters should carefully discuss regulations and their application with Chinese importers to ensure that their interpretation is accurate.

Includes PSD changes: No
Includes Trade Matrix: No
Unscheduled Report
Beijing [CH1], CH

This report was prepared by the Office of Agricultural Affairs of the USDA/Foreign Agricultural Service in Beijing, People's Republic of China for U.S. exporters of domestic food and agricultural products. While every possible care was taken in the preparation of this report, information provided may not be completely accurate either because policies have changed since its preparation, or because clear and consistent information about these policies was not available. It is highly recommended that U.S. exporters verify the full set of import requirements with their foreign customers, who are normally best equipped to research such matters with local authorities, before any goods are shipped. FINAL IMPORT APPROVAL OF ANY PRODUCT IS SUBJECT TO THE IMPORTING COUNTRY'S RULES AND REGULATIONS AS INTERPRETED BY BORDER OFFICIALS AT THE TIME OF PRODUCT ENTRY.

Summary

This standard details the technical requirements for testing, hygiene, labeling, packaging, and storage requirements for fresh and frozen poultry products. The Ministry of Health has statutory responsibility for this standard. The following material was adopted on April 1, 2002 for implementation from September 30, 2002. The Regulation was published by the Standardization Administration of the People's Republic of China.

BEGIN TRANSLATION

Fresh and Frozen Poultry Product Regulation (GB16869-2002)

This standard is mandatory except for Chapter 6 which is voluntary. This standard is a revised version of GB 16869-2000. The main revisions made are as follows:

- Nullifies "methamidophos, no more than 0.02mg/kg", "clenbuterol hydrochloride, zero content", and Appendix B;
- Adds "congestion area less than 0.5cm² not counted", "calculation and testing methodologies of congestions and hard feathers";
- The core temperature of frozen poultry products has been adjusted from no higher than minus 15 degrees Celsius to no higher than minus 18 degrees Celsius;
- Defrost water loss rate has been adjusted from no more than 8% to no more than 6%;
- Lead limitation has been adjusted from no more than 0.5mg/kg to no more than 0.2mg/kg;
- BHC limitation has been adjusted from no more than 0.2 mg/kg to no more than 0.1 mg/kg;
- E. Coli limitations in frozen poultry products have been adjusted from 1x10³zmpn/100g to 5x10³MPN/100g;
- Diethylstilbestrol will be determined in accordance with SN 0672-1997 Determination of Diethylstilbestrol Residue in Export Meat and Meat Products-Radiation Immunization Method, instead of GB/T 14931.2-1994 Determination of Diethylstilbestrol in Poultry Products;
- Routine inspections, sampling design for inspection, and allowable quantity of defects are adopted in line with testing standard I and standard II stipulated in CAS RM 42-1969 Sampling Design for Pre-packaged Foods.

GB 16869-2000 will be annulled upon implementation of this standard.

Appendix A is a part of this standard.

This standard is jointly proposed by the National Committee of Standardization Technology for Food Industry and the Technical Committee of Food Hygiene Standard under the Hygiene Standard Technical Committee of the Ministry of Health.

This standard is drafted by the following agencies: Institute of Supervision and Inspection on Food Hygiene under the Ministry of Health, Secretariat of National Committee on Standardization Technology for Food Industry, and Institute of Hygiene Supervision of Shanghai Health Bureau. Also participated in the drafting process are Assessment Center of Slaughtering Technology under Domestic Trade Administration, Livestock and Poultry Product Quality Inspection Center of Ministry of Agriculture, China Meat Association, Beijing Bureau of Exit-Entry Inspection and Quarantine, and Shenzhen Bureau of Exit-Entry Inspection and Quarantine.

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The appendix is drafted by the Institute of Nutrition and Food Hygiene under the Chinese Academy of Preventive Medicine and the Institute of Supervision and Inspection on Food Hygiene under the Ministry of Health.

Persons drafting Appendix A are Chen Huijing, Wang Xuqing, Yang Dajin, and Wu Guohua.

Fresh and Frozen Poultry Product National Standard of the People's Republic of China
GB 16869-2002 (replaces GB 16869-2000)

Chapter One: Scope

This standard stipulates the definition, testing methods, testing rules, labeling, packaging, and storage requirements for fresh and frozen poultry products.

This standard applies to fresh or frozen poultry products derived from healthy and live poultry that have been slaughtered, processed and packaged.

Chapter Two: Referenced Standards

The clauses in the below standards, though referenced in this standard, have become clauses of this standard. All listed documents are applicable at the time of publication of this standard. However, all standards are subject to modification and the parties that apply this standard should study the possibility of using the recent versions of the following standards.

GB 191-2000 Packaging, Labeling, Storage and Transportation

GB4789.2-1994 Microbial Analysis of Food Hygiene Detection of Colony Numbers

GB 4789.3-1994 Microbial Analysis of Food Hygiene Detection of Escherichia Coli

GB 4789.4-1994 Microbial Analysis of Food Hygiene Detection of Salmonella

GB 4789.6-1994 Microbial Analysis of Food Hygiene Detection of Enterorrhea Escherichia Coli

GB/T 5009.11-1996 Determination of Arsenic in Food.

GB/T 5009.12-1996 Determination of Lead in Food.

GB/T 5009.17-1996 Determination of Mercury in Food.
GB/T 5009.19-1996 Determination of BHC and DDT Residues in Food.
GB/T 5009.44-1996 Analysis Methods of Hygiene Standards in Meat and Meat Products.
GB/T 6388-1996 Marking for Transportation, Packaging, Product Distribution.
GB 7718-1994 General Standard for Food Labeling.
GB/T 14931.1-1994 Determination of Oxytetracycline, Tetracycline and Chlortetracycline Residue Levels in Poultry Meat (HPLC).
SN/T 0208-1993 Determination of Ten Types of Sulfanilamide in Meat Exports.
SN/T0212.3-1993 Determination of Clopidol in Poultry Meat Exports (Propionylation-Gas Chromatography)
SN 0672-1997 Determination of Diethylstilbestrol Residue in Exported Meat and Meat Products (Radiation Immunization)

Chapter Three: Terminology

This standard adopts the following definitions:

3.1 Poultry product

All edible portions including entire poultry carcass, disjointed parts (muscle, wing, leg, etc), and by-products (head, neck, intestine, claw), after live poultry are slaughtered and processed.

3.2 Fresh poultry product

Chilled fresh product, not frozen, but subjected to low temperatures after live poultry are slaughtered and processed.

3.3 Frozen poultry product

Frozen treated product with core temperature below minus 18 degrees Celsius after live poultry are slaughtered and processed.

3.4 Hard feather

Feathers having a length of 12 mm or feather roots with a diameter of more than 2 mm.

3.5 Visible foreign matter

Any inedible exotic or contaminate matter in product (e.g. yellow skin, excrement, bile, and non-poultry origin contaminates; such as plastic, metal or feed residue).

Chapter Four: Technical Requirements

4.1 Material

Live poultry shall come from epidemic-free areas and be subject to quarantine.

4.2 Processing

Poultry should be quarantined and certified before processing.

4.2.1 Separating

Poultry should be treated in low temperature before separation. The time between slaughter and placement into cold storage shall not last more than 2 hours.

4.2.2 Post-processing treatment

After separation, every poultry part shall be clipped including trauma site, blood spots, blood contaminations, feather roots, etc.

4.3 Frozen processing

The core temperature for products that require freezing should reach minus 15 degrees within 12 hours.

4.4 Physical property

Shall comply with stipulations of Table 1.

Table 1

| Item | Fresh poultry products | Frozen poultry products (after defrosting) |
|---|---|---|
| Tissue feature | The muscle is elastic and any point of pressure recovers immediately. | Point of pressure on the muscle recovers more slowly and does not recover completely. |
| Color | The surface and muscle possess the intrinsic sheen of poultry. | |
| Flavor | Possesses the intrinsic odor of poultry, without any foreign odor. | |
| Boiled broth | Hyaline with fatty gathering on the surface of broth and possesses intrinsic taste. | |
| Congestion (use "S" as congestion area, in cm ²) S > 1 0.5 < S # 1 S # 0.5 | Must not be detected Less than 2% of the sample Not counted | |
| Hard feather (piece/10kg) | # 1 | |
| Visible foreign matter | Must not be detected | |

Note: Congestion area is calculated on the basis of one piece of congestion in a whole poultry carcass or in a separated part.

4.5 Physical and chemical reference

Both fresh and frozen poultry products shall comply with the stipulations of Table 2.

Table 2

| Item | Reference |
|--|--|
| Defrost water loss rate, % | # 6 |
| Volatility of saline nitrite, mg/100g | # 15 |
| Mercury (Hg), mg/kg | # 0.05 |
| Lead (Pb), mg/kg | # 0.2 |
| Arsenic (As), mg/kg | # 0.5 |
| BHC, mg/kg Tests from sample if fat content is less than 10% Tests from fat if fat content is no less than 10% | # 0.1 # 1 |
| DDT, mg/kg Tests from sample if fat content is less than 10% Tests from fat if fat content is no less than 10% | # 0.2 # 2 |
| Dichlorvos, mg/kg | # 0.05 |
| Tetracycline, mg/kg | # 0.25 (muscle) # 0.3 (liver) # 0.6 (kidney) |
| Chlortetracycline, mg/kg | # 1 |
| Oxyletracycline, mg/kg | < 0.1 (muscle) # 0.3 (liver) # 0.6 (kidney) |
| Sulfadimidine, mg/kg | # 0.1 |
| Clopidol, mg/kg | # 0.01 |
| Diethylstilbestrol | Must no be detected |

4.6 Microorganism reference

Shall comply with stipulated in Table 3.

Table 3

| Item | Reference | |
|------------------------------|-----------------------|------------------------|
| | Fresh poultry product | Frozen poultry product |
| Colony number, cfu/g | # 1×10^6 | # 5×10^5 |
| E.Coli, MPN/100g | < 1×10^4 | < 5×10^3 |
| Salmonella | Must not be detected | |
| Enterorrhea Escherichia coli | Must not be detected | |

Chapter Five: Detection Methods

5.1 Sensory properties

Frozen poultry products should be assessed after defrosting.

5.1.1 Tissue status, color, flavor

After a microorganism test sample has been taken, place the sample material under natural light or in an equivalent circumstance and detect tissue status by touching, color by sighting, and odor by smelling.

5.1.2 Boiled broth

Mince sample (6.5.4), weigh 20 grams, place in 200mL beaker, add 100mL water, cover the beaker, heat it until 50-60 degrees Celsius, remove the cover, detect the odor by smelling; boil it and assess the status of boiled broth and fat coagulation. Taste the boiled broth after it cools.

5.1.3 Congestion

Measure congestion area through appropriate means after assessment of tissue status, color, and flavor. The number of congestions with an area of more than 0.5 cm² but no more than 1 cm² is determined through Equation (1):

$$X = A1/A * 100$$

where X = ratio of the number of congestions ($0.5 \text{ cm}^2 < S \leq 1 \text{ cm}^2$) against the total number of poultry products (entire poultry, poultry parts, legs or wings, are counted in pieces, the same below);

A = the total number of poultry products in a basic unit;

A1 = the number of congestions ($0.5 \text{ cm}^2 < S \leq 1 \text{ cm}^2$) in a basic unit

5.1.4 Hard feather

Done simultaneously with assessment of tissue status, color, and flavor by using vernier caliper with a precision of 0.05 mm as in Equation (2):

$$X1 = A2/W * 10$$

where X1 = the number of hard feathers in every 10 kilograms in a basic unit;

A2 = the actual number of hard feathers in a basic unit;

W = mass of sample in a basic unit, kg.

5.1.5

Visible foreign matter

Done simultaneously with assessment of tissue status, color, and flavor by using visual means

5.2

Defrost water loss

5.2.1 Apparatus and instrument

Electrical scale: sensitivity (1g); Thermometer: range of minus 10 to minus 50 degrees and graduation of 0.5 degrees; Porcelain disc and barbed wire.

5.2.2 Test procedures

Place wire mesh on porcelain disc. The distance between the bottom of the porcelain disc and the wire mesh shall be greater than 2 cm. Pick between 1000 g to 1200 g meat from the sample, weigh the sample on the electrical scale and place it on the wire mesh. Use plastic film to cover the sample and allow the product to defrost at a natural temperature between 15 degrees and 25 degrees. When the core temperature reaches 2 degrees to 3 degrees, weigh the sample again. Place the sample on the wire mesh for 30 min and weigh the sample. Repeat the previous step until the deviation between the two weights does not surpass 2 g.

5.2.3 Description of determination result

Defrost water loss rate of sample is calculated by Equation. (3):

$$X2 (\%) = ((m - m1) / m) * 100$$

Where X2 = defrost water loss ratio of sample, %;

m = mass of the sample before defrosting, g;

m1 = mass of the sample after defrosting, g.

Calculated result should be rounded to an integer number.

5.3 Volatile saline nitrite

Determined by methodology prescribed in provision 4.1 of GB/T 5009.44-1996.

5.4 Mercury

Determined by methodology prescribed in GB/T 5009.17.

5.5 Arsenic

Determined by methodology prescribed in GB/T 5009.11.

5.6 Lead

Determined by methodology prescribed in GB/T 5009.12.

5.7 BHC, DDT

Determined by methodology prescribed in GB/T 5009.19

5.8 Dichlorvos

Determined by methodology prescribed in Appendix A of this standard.

5.9 Tetracycline, Chlortetracycline, Oxyletracycline

Determined by methodology prescribed in GB/T 14931.1.

5.10 Sulfadimidine

Determined by methodology prescribed in SN/T 0208.

5.11 Clopidol

Determined by methodology prescribed in SN/T 0212.3.

5.12 Diethylstilbestrol

Determined by methodology prescribed in SN 0672.

5.13 Colony number

Tested by methodology prescribed in GB 4789.2.

5.14 E.Coli

Tested by methodology prescribed in GB 4789.3.

5.15 Salmonella

Tested by methodology prescribed in GB 4789.4.

5.16 Enterorrhea Escherichia coli

Tested by methodology prescribed in GB 4789.6.

5.17 Product core temperature

5.17.1 Thermometer

Non-mercury glass thermometer with a range of minus 20 to minus 50 degrees Celsius, or other types of temperature measuring devices.

5.17.2 Test procedures

Using a bore with a diameter larger than the thermometer, drill into the muscle core, remove the bored sample, and immediately insert the non-mercury thermometer into the muscle core. Read the temperature after it stabilizes.

Chapter Six: Inspection rules

6.1 Inspection classification

6.1.1 Routine inspection

Routine inspection shall be conducted in one of the following situations:

- a) an isolated batch of product submitted for inspection;
- b) change in location of live poultry production;
- c) inaugural processing of a new plant;
- d) processing for a consecutive of 6 months, or resumption of processing after a halt (suspension);
- e) result of acceptance inspection significantly differs from that of previous routine inspection;
- f) Requested by a quality or hygiene monitoring agency.

6.1.1.2 Routine inspection covers items prescribed in Table 1, Table 2, and Table 3 of this

standard

6.1.2 Acceptance inspection

6.1.2.1 Acceptance inspection shall be conducted for all ex-factory products.

6.1.2.2 Acceptance inspection includes items listed in Table 1, defrost water loss rate of frozen poultry products, volatile saline nitride, colony number, and E. Coli.

6.2 Group batch

Determined by provisions 6.2.1 or 6.2.2.

6.2.1 Consecutive batch

Products of identical processing conditions, identical part (entire poultry carcass, muscle, wing, head, claw, organs), identical package, and delivered at one time constitute a consecutive batch. Batch quantity is calculated by the number of basic packaging containers (hereinafter referred to as "basic container").

6.2.2 Isolated batch

An isolated batch is defined by products from an identical part, identical package, and submitted for inspection at one time. Batch quantity is calculated by the number of basic containers.

6.3 Sampling

6.3.1 Routine sampling inspection

Based on batch quantity, samples are randomly taken according to sampling number prescribed in Table 4;

Table 4

| Batch quantity (basic container) | Sampling number (basic container) | Allowable number of defects (basic container) |
|----------------------------------|-----------------------------------|---|
| 600 or less than 600 | 13 | 2 |
| 601-2000 | 21 | 3 |
| 2001-7200 | 29 | 4 |
| 7201-15000 | 48 | 6 |
| 15001-24000 | 84 | 9 |
| 24001-42000 | 126 | 13 |
| more than 42000 | 200 | 19 |

6.3.2 Sampling of acceptance inspection

Based on batch quantity, samples are randomly taken according to the sampling number prescribed in Table 5.

Table 5

| Batch quantity (basic container) | Sampling number (basic container) | Allowable number of defects (basic container) |
|----------------------------------|-----------------------------------|---|
| 600 or less than 600 | 6 | 1 |
| 601-2000 | 13 | 2 |
| 2001-7200 | 21 | 3 |
| 7201-15000 | 29 | 4 |
| 15001-24000 | 48 | 6 |
| 24001-42000 | 84 | 9 |
| more than 42000 | 126 | 13 |

6.4 Sample taking procedures and inspection procedures

Entire sample of fresh poultry product

•

Take microorganism test sample

▼

Microorganism Test

•

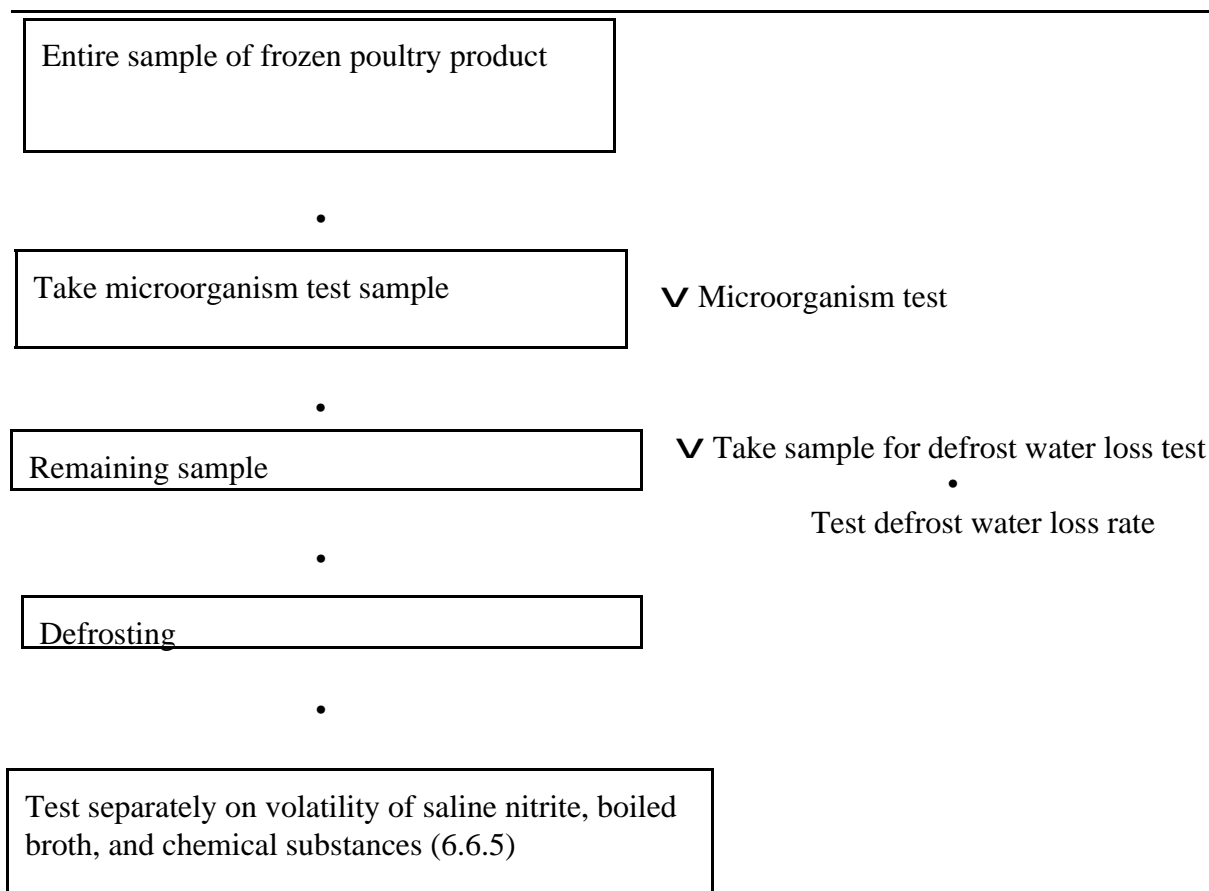
Remaining sample

•

Test tissue status, color, flavor, hard feather, congestion, visible foreign matter

•

Test separately on volatility of saline nitrite, boiled broth, and chemical substances (6.6.5)



6.5 Sample taking methodology

The following sample shall not contain congestion, hard feather, or visible foreign matter.

6.5.1 Microorganism test sample

Randomly pick three basic containers of samples from all sampling material, take 100 grams of samples from each container under aseptic handling procedures and mix them.

6.5.2 Test sample for defrost water loss

Randomly pick 3-5 basic containers of samples from all sampling materials, take 500 grams of samples from each container, mix and place them into an insulated container.

6.5.3 Test sample for volatility of saline nitrite

Randomly pick three basic containers of samples from all sampling materials, take 100 grams of samples, free from fat and bones, from each container and mix them up.

6.5.5 Test sample for chemical substances (12 types listed in Table 2 of this standard, such as mercury, diethylstilbestrol, etc.).

Randomly pick three basic containers of samples from all sampling materials, take 200 grams of edible parts and mix them.

6.6 Assessment rules and proof-test

6.6.1 Defects classification

6.6.1.1 General defect

Refers to congestions and hard feathers not in compliance with this standard.

6.6.1.2 Serious defect

Refers to tissue status, color, flavor, boiled broth, and items stipulated in Table 2 and Table 3 do not meet this standard, or there are visible foreign matters.

6.6.2 Determination of all test results

6.6.2.1 Determination of result of congestions and hard feathers

The result of congestion and hard feather is determined on the basis of one basic container.

Example 1:

There are 6 basic containers of samples, number them.

Test result: congestions in container No. 1 and hard feathers in container No. 3 do not comply with this standard.

Conclusion: two containers have general defects.

Example 2:

There are 13 basic containers of samples, number them.

Test result: hard feathers in containers No. 1 through No. 13 are not compliant with this standard, while congestions in No. 8 container do not comply with this standard.

Conclusion: the 13 basic containers have general defects.

6.6.2.2 Determination of test result for tissue status, color, flavor, boiled broth, and items listed in Table 1, and Table 2 of this standard.

If test results of any item do not comply with this standard, the entire sample is considered to have serious defects.

6.6.3 Determination of routine inspection and proof-test

6.6.3.1 If all test items (6.1.1.2) meet this standard, the whole batch of products is considered up to standard.

6.6.3.2 If any item has been tested as having serious defect (6.6.1.2), the entire batch of products is considered not qualified and no proof-test is needed.

6.6.3.3 If the general defects (6.6.1.1) of routine inspection do not exceed the allowable number stipulated in Table 4, the entire batch is considered up to standard. If the general defects exceed the allowable number of Table 4, a proof-test is needed to repeat the items in Table 4. Based on the proof-test result and allowable general defects of Table 4, a qualified or unqualified determination is offered.

6.6.4 Determination of acceptance inspection and proof-test

6.6.4.1 If all tests of acceptance inspection (6.1.2.2) meet this standard, the entire batch of products is considered up to standard.

6.6.4.2 If any item of acceptance inspection has been tested as having serious defects (6.6.1.2), the entire batch of products is considered unqualified and no proof-test is needed.

6.6.4.3 If the general defects (6.6.1.1) of acceptance inspection do not exceed the allowable number as stipulated in Table 5, the entire batch of products is considered up to standard; if it exceeds the allowable number on Table 5, a proof-test is needed to repeat the items in Table 5. A qualified or unqualified determination will be offered based on the result of the result of the proof-test and allowable number of general defects.

7 Labeling, marking, packaging, and storage

7.1 Labeling and marking

7.1.1 Labeling

Labeling of pre-packaged poultry products shall comply with stipulations in GB 7718.

7.1.2 Package marking in transportation

Package marking and indicators of consignee and consignor shall comply with stipulations of GB 191 and GB/T 6388.

7.2 Packaging

Packaging is required for both fresh and frozen poultry products, and new and sanitation-compliant packaging materials shall be used.

7.3 Storage

Frozen poultry products shall be placed in cold storage with a temperature below minus 18 degrees Celsius, and the variation of storage temperature in 24 hours shall not exceed one degree Celsius.

Appendix A (Appendix to the standard)

Determination of multi-constitutional organic phosphorus residue in animal based foods

This appendix stipulates the detection of multi-constitutional organic phosphorus residue; including organic phosphorus methamidophos, dichlorvos, acephate, monocrotophos, dimethoate, disulfaton, methyl-parathion, fenitrothion, pirimiphos methyl, Malathion, fenthion, parathion, ethion, in poultry meat, milk, milk product, egg, and egg product

Minimum detection (: g/kg): Methamidophos (5.7), didchlorvos (3.5), acephate (10.0), monocrotophos (12.0), dimethoate (2.6), disulfaton (1.2), methyl-parathion (2.6), fenitrothion (2.9), pirimiphos methyl (2.5), Malathion (2.8), fenthion (2.1), parathion (2.6), ethion (1.7).

A1 Summary of Methodology

Samples are taken using extraction, purification, concentration, volume, and separation (capillary column gas chromatography) are detected by flame photometer detector, and described using retention time, and quantified using extensional method.

Sequence of peaks: methamidophos, dichlorvos, acephate, monocrotophos, dimethoate, disulfaton, methyl-parathion, fenitrothion, pirimiphos methyl, malathion, fenthion, parathion, ethion.

A2 Reagents

Water used in this experiment shall be prepared according to the second grade standard of GB/T 6682. The reagents used in this experiment shall all be analytical reagent grade chemicals except for the following;

A2.1 Acetone: redistillation

A2.2 Dichloromethane: redistillation

A2.3 Acetic ether: redistillation

A2.4 Cyclohexane: redistillation

A2.5 Sodium chloride.

A2.6 Anhydrous Sodium sulfate

A2.7 Gel: Bio-Beads S-X3 (or another gel that has the same function as Bio-Beads S-X3); 200-400 mesh

A2.8 All the following organic phosphorus standard product contents, including: methamidophos, dichlorvos, acephate, monocrotophos, dimethoate, disulfaton, methyl-parathion, fenitrothion, methyl, Malathion, fenthion, parathion, ethion, shall not be less than 99 percent.

A2.9 Preparation of organic phosphorus standard solution

A2.9.1 Preparation of a single item organic phosphorus standard solution: Weigh 0.0100g of organic phosphorus and place into a 25ml volumetric flasks. Dissolve in ecetic ether solution and determine the final volume (having concentration of 400 : g/ml).

A2.9.2 Preparation of a mixed organic phosphorus standard solution: before testing, place separate volumes of a single item organic phosphorus standard solution (using A2.9.1) into 10ml volumetric flasks, respectively; blow away the solvent using nitrogen gas, dilute it with a fresh milk extraction prepared by means of A5.1.3 (extraction) and A5.2 (purification) and determine the final volume. The concentration (: g/ml) of each organic phosphorus from the mixture is: Methanidophos (16), didchlorvos (80), acephate (24), monocrotophos (80), dimethoate (16), disulfaton (24), parathion (16), fenitrothion (16), pirimiphos methyl (16), Malathion (16), fenthion (24), parathion (16), ethion (8).

NOTE: If only dichlorvos is being tested, a standard storage solution and standard application solution shall be prepared.

A3 Apparatus and Equipment

A3.1 Chromatography-gas: with flame photometer detector (FPD-P) and capillary chromatogram column.

A3.2 Revolving-evaporate vessel

A3.3 Gel-depurator: length: 30cm; internal diameter: 2.5cm; having piston-glass-chromatography column which has a little amount of glass cotton on the bottom. Place gel, that has been soaked in the eluant of acetic ether : cyclohexane (1:1), into the column (wet method). Column height is 26cm and gel bed shall be kept in the eluant.

A4 Preparation of samples

Egg and egg product: peeled and homogenized.

Meat and meat product: cut into slice after deboning and made into meat paste.

Milk and mild product: homogenized.

A5 Analysis Steps

A5.1 Extraction, Distribution, and Concentration

A5.1.1 Egg and egg product: weigh 20.0 g (precision to 0.01 g) sample and place into a 100ml stoppered flask. Add 5 ml water (add water, according to the water content of the sample, to make the final water volume 20 ml). Add 40ml Acetone to the flask and shake for 30 minutes. Add 6g Sodium chloride and shake thoroughly, then add 30ml Dichloromethane and shake for 30 minutes. Absorb 35ml supernatant and concentrate using revolving-vaporizer with Anhydrous Sodium sulfate to 1 ml. Add 2 ml acetic ether and cyclohexane solution and concentrate again. Repeat the above step 3 times and concentrate to 1 ml.

A5.1.2 Meat and meat product: weigh 20.0 g (precision to 0.01 g) sample and place into a 100 ml stoppered flask. Add 6 ml water (precision to 0.01g) to the flask and proceed with the steps of A5.1.1.

A5.1.3 Milk and milk product: weigh 20.0 g (precision to 0.01 g) sample and place into a 100 ml stoppered flask and proceed with the steps of A5.1.1

A5.2 Purification

Make the prepared concentration using gel-depurator and dilute with acetic ether and cyclohexane solution. Remove the first 35 ml portion and collect the 35 ml to 70 ml portion and concentrate this portion to 1 ml by revolving-vaporizer. Purify using gel-depurator and collect the 35 ml to 70 ml fraction and concentrate this to 1 ml by revolving-vaporizer. Transfer the concentration into a 5 ml tube, wash the revolving-evaporation vessel several times with 5 ml acetic ether and transfer the wash into the same tube. Blow the liquid to below 1 ml with Nitrogen gas and add acetic ether to make the final volume read 1 ml for chromatographic analysis.

A5.3 Chromatographic condition

A5.3.1 Chromatogram column: elastic quartz capillary column with an internal diameter of 0.32 mm and a length of 30mm, spread with SE-54 (thickness of 0.25 mm)

A5.3.2 Column temperature: temperature programming
60 degrees (at a rate of 40 degrees per minute) to 110 degrees (at a rate of 5 degrees per minute)
to 235 degrees (at a rate of 40 degrees per minute) to 265 degrees

A5.3.3 Inlet temperature: 270 degrees.

A5.3.4 Detector: flame photometer detector (FPD-P), temperature 270 degrees.

A5.3.5 Carrier gas: Nitrogen gas, velocity 1 mL/min, blow velocity 50 mL/min.

A5.3.6 Hydrogen and air velocity: Hydrogen 50mL/min, air 500mL/min.

A5.4 Detection

Weigh 1: L mixed solution of organic phosphorus and purified solution and insert it into the chromatogram. Describe using retention time and comparative values for the high peak or peak area of sample and applicable standard for solution.

A5.5 13 types of organic phosphorus chromatograms

(FIG. A1) See Chinese version.

A6 Description of analysis

The residue of some organic phosphorus in the sample is calculated by Equation (A1):

$$X = (m1 * V2 * 1000) / (m * V1 * 1000) = (m1 * V2) / (m * V1) \dots\dots\dots(A1)$$

where X = some organic phosphorus residue in the sample, mg/kg;

m = the mass of the sample, g;

m1 = the organic phosphorus residue in the preparation, ng;

V1 = the inlet volume, : L;

V2 = the final volume of the preparation, mL.

A7 Allowable deviation

The deviation from values of two experiments on the same sample shall not surpass 20 percent of the average value.

A8 Accuracy

Accuracy is valued by recovery rate. According to requirements, add some kind of organic phosphorus standard solution to poultry meat, eggs, or milk, and test the recovery rate which should vary from 70% to 110%. The recovery rate is calculated by Equation (A2):

$$Y = ((m1 - m2) / m) * 100 \dots\dots\dots(A2)$$

where Y = the recovery rate, %;

m1 = the dictation constituent i of the sample after adding standard solution;

m2 = the constituent i content of the sample;

m = the value of the constituent i.

END TRANSLATION